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Multi-Media, Multi-Concentration Chlorinated Biphenyl Congeners Analytical Service for Superfund (CBC01.0)

Office of Superfund Remediation and Technology Innovation (OSRTI)
Analytical Services Branch (ASB) (5102G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites are a series of Chlorinated Biphenyl (CB) Congeners that are analyzed using High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS). The Analytical Services Branch (ASB) of EPA's Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water, soil, sediment, sludge, non-human tissue, ash, oil, and oily matrices for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the CB congeners analytical service produces data of known and documented quality.

DESCRIPTION OF SERVICES

The CB congeners analytical service provides a flexible contractual framework for laboratories to apply EPA analytical methods for the isolation, detection, and quantitative measurement of 209 2,2',3,3',4,4',5,5',6,6'-substituted mono- through deca- CB congeners in water, soil, sediment, sludge, non-human tissue, ash, oil, and oily matrices. EPA ASB has prequalified laboratories that use the CB congeners Statement of Work (SOW) CBC01.0 to provide this service. The standard data Turnaround Time (TAT) for this service is 35 days after laboratory receipt of the last sample in the Sample Delivery Group (SDG). This TAT can be changed to meet project-specific requirements.

REQUESTING THIS FLEXIBLE SERVICE

The CB congeners analytical service can be requested by EPA Regions and other interested parties by submitting a Task Order (TO) to EPA ASB. These TOs can modify the SOW to meet project-specific requirements [e.g., changes in TAT, detection limits, or Target Compound Lists (TCLs)]. The CB congeners SOW can be accessed at:

http://www.epa.gov/superfund/programs/clp/nonroutine.htm

DATA USES

The CB congeners analytical service provides data that EPA uses for a variety of purposes such as: determining the nature and extent of contamination at a hazardous waste site; assessing priorities for response based on risks to human health and the environment; determining appropriate clean-up actions; and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections; Hazard Ranking System (HRS) scoring; remedial investigation/ feasibility studies; remedial design; treatability studies; and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

The applicable target compounds and Contract Required Quantitation Limits (CRQLs) for this service are listed in **Table 1**. For water samples, the lowest reportable CRQL is 10 pg/L. For soil and tissue samples, the lowest reportable CRQL is 1.0 ng/kg. For extracts, the lowest reportable CRQL is 0.5 pg/µL. The specific CRQLs are highly matrix-dependent. The CRQLs listed herein are provided for guidance and may not always be achievable.

METHODS AND INSTRUMENTATION

For water samples, the stable isotopically-labeled analogs of the Toxics and labeled Level of Chlorination (LOC) CB congeners are spiked into a 1 L sample. The sample is extracted using solid-phase extraction (SPE), separatory funnel extraction (SFE), or continuous liquid/liquid extraction (CLLE).

For soil/sediment samples, the labeled compounds are spiked into a sample containing 10 g (dry weight) of solids and extracted for 16-24 hours with toluene in a Soxhlet/Dean-Stark (SDS) extractor. The extract is concentrated for cleanup.

For non-human tissue samples, a 20 g aliquot of a sample is homogenized, and a 10 g aliquot is spiked with the labeled compounds. The sample is mixed with anhydrous sodium sulfate, allowed to dry for 12-24 hours, and extracted for 18-24 hours using methylene chloride in an SDS extractor. The extract is evaporated to near dryness, and the lipid content is determined.

For all samples, the extracts are cleaned and concentrated to 20 µL. Labeled internal standards are added to each extract and an aliquot of the extract is injected into the High Resolution Gas Chromotograph (HRGC). The analytes are separated by the HRGC and detected by a High Resolution Mass Spectrometer (HRMS). **Table 2** summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for the CB congeners analytical service include the hardcopy data reporting forms and supporting raw data. Certain TOs may require the use of the Electronic Data Deliverable (EDD) format. The EDD reporting requirements will be outlined in the TO. The laboratory must submit data to EPA within 35 days after laboratory receipt of the last sample in the SDG, or as stated in the TO. The EPA Regions then review the data, based on project-specific requirements.

QUALITY ASSURANCE (QA)

The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of the quality required.

During the *planning* of an environmental data collection program, QA activities focus on defining data quality criteria and designing a Quality Control (QC) system to measure the quality of data being generated. During the *implementation* of the data collection effort, QA activities ensure that the QC system is functioning effectively, and the deficiencies uncovered by the QC system are corrected.

After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA/QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL (QC)

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analysis are shown in **Table 3**. A number of optional cleanup procedures are available for the CB congeners SOW.

Table 1. Target Compound List (TCL) and Estimated Contract Required Quantitation Limits (CRQLs)

CB Congener[1]	IUPAC Number	Water (pg/L)	Other (ng/kg)	Extract (pg/µL)
Monochlorobiphenyls				
2-MoCB	1	200	20	10
3-MoCB	2	10	1	0.5
4-MoCB	3	200	20	10
Dichlorobiphenyls				
2,2'-DiCB	4	500	50	25
2,3-DiCB	5	50	5	2
2,3'-DiCB	6	50	5	2
2,4-DiCB	7	50	5	2
2,4'-DiCB[2]	8	500	50	25
2,5-DiCB	9	50	5	2
2,6-DiCB	10	50	5	2
3,3'-DiCB	11	200	20	10
3,4-DiCB	12	100	10	5
3,4'-DiCB	13	100	10	5
3,5-DiCB	14	100	10	5
4,4'-DiCB	15	500	50	25
Trichlorobiphenyls				
2,2',3-TrCB	16	100	10	5
2,2',4-TrCB	17	200	20	10
2,2',5-TrCB[2]	18	500	50	25
2,2',6-TrCB	19	100	10	5
2,3,3'-TrCB	20	500	50	25
2,3,4-TrCB	21	200	20	10
2,3,4'-TrCB	22	200	20	10
2,3,5-TrCB	23	200	20	10
2,3,6-TrCB	24	200	20	10
2,3',4-TrCB	25	200	20	10
2,3',5-TrCB	26	200	20	10
2,3',6-TrCB	27	200	20	10
2,4,4'-TrCB[2]	28	500	50	25
2,4,5-TrCB	29	200	20	10
2,4,6-TrCB	30	500	50	25
2,4',5-TrCB	31	500	50	25
2,4',6-TrCB	32	200	20	10
2',3,4-TrCB	33	200	20	10
2'3,5-TrCB	34	200	20	10
3,3',4-TrCB	35	200	20	10
3,3',5-TrCB	36	200	20	10
3,4,4'-TrCB	37	500	50	25
3,4,5-TrCB	38	200	20	10
3,4',5-TrCB	39	200	20	10
Tetrachlorobiphenyls				
2,2',3,3'-TeCB	40	500	50	25
2,2',3,4-TeCB	41	500	50	25
2,2',3,4'-TeCB	42	200	20	10
2,2',3,5-TeCB	43	200	20	10
2,2',3,5'-TeCB[2]	44	500	50	25
2,2',3,6-TeCB	45	200	20	10
2,2',3,6'-TeCB	46	200	20	10

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) Con't

CB Congener[1]	IUPAC Number	Water (pg/L)	Other (ng/kg)	Extract (pg/µL)
2,2',4,4'-TeCB	47	500	50	25
2,2',4,5-TeCB	48	200	20	10
2,2',4,5'-TeCB	49	500	50	25
2,2',4,6-TeCB	50	200	20	10
2,2',4,6'-TeCB	51	200	20	10
2,2',5,5'-TeCB[2]	52	500	50	25
2,2',5,6'-TeCB	53	200	20	10
2,2',6,6'-TeCB	54	500	50	25
2,3,3',4'-TeCB	55	500	50	25
2,3,3',4'-TeCB	56	200	20	10
2,3,3',5-TeCB	57	500	50	25
2,3,3',5'-TeCB	58	500	50	25
2,3,3',6-TeCB	59	200	20	10
2,3,4,4'-TeCB	60	500	50	25
2,3,4,5-TeCB	61	500	50	25
2,3,4,6-TeCB	62	200	20	10
2,3,4',5-TeCB	63	500	50	25
2,3,4',6-TeCB	64	200	20	10
2,3,5,6-TeCB	65	500	50	25
2,3',4,4'-TeCB[2]	66	500	50	25
2,3',4,5-TeCB	67	500	50	25
2,3',4,5'-TeCB	68	500	50	25
2,3',4,6-TeCB	69	500	50	25
2,3',4',5-TeCB	70	500	50	25
2,3',4',6-TeCB	71	500	50	25
2,3',5,5'-TeCB	72	500	50	25
2,3',5',6-TeCB	73	500	50	25
2,4,4',5-TeCB	74	500	50	25
2,4,4',6-TeCB	75	200	20	10
2',3,4,5-TeCB	76	500	50	25
3,3',4,4'-TeCB [2,3]	77	500	50	25
3,3',4,5-TeCB	78	500	50	25
3,3',4,5'-TeCB	79	500	50	25
3,3',5,5'-TeCB	80	500	50	25
3,4,4',5-TeCB[3]	81	500	50	25
Pentachlorobiphenyls				
2,2',3,3',4-PeCB	82	500	50	25
2,2',3,3',5-PeCB	83	500	50	25
2,2',3,3',6-PeCB	84	500	50	25
2,2',3,4,4'-PeCB	85	200	20	10
2,2',3,4,5-PeCB	86	500	50	25
2,2',3,4,5'-PeCB	87	500	50	25
2,2',3,4,6-PeCB	88	500	50	25
2,2',3,4,6'-PeCB	89	500	50	25
2,2',3,4',5-PeCB	90	1000	100	50
2,2',3,4',6-PeCB	91	500	50	25
2,2',3,5,5'-PeCB	92	500	50	25
2,2',3,5,6-PeCB	93	500	50	25
2,2',3,5,6'-PeCB	94	500	50	25

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) Con't

CB Congener[1]	IUPAC Number	Water (pg/L)	Other (ng/kg)	Extract (pg/µL)
2,2',3,5',6-PeCB	95	500	50	25
2,2',3,6,6'-PeCB	96	500	50	25
2,2',3',4,5-PeCB	97	500	50	25
2,2',3',4,6-PeCB	98	500	50	25
2,2',4,4',5-PeCB	99	500	50	25
2,2',4,4',6-PeCB	100	500	50	25
2,2',4,5,5'-PeCB[2]	101	1000	100	50
2,2',4,5,6'-PeCB	102	500	50	25
2,2',4,5,'6-PeCB	103	500	50	25
2,2',4,6,6'-PeCB	104	500	50	25
2,3,3',4,4'-PeCB[2,3]	105	200	20	10
2,3,3',4,5-PeCB	106	500	50	25
2,3,3',4',5-PeCB	107	1000	100	50
2,3,3',4,5'-PeCB	108	500	50	25
2,3,3',4,6-PeCB	109	200	20	10
2,3,3',4',6-PeCB	110	1000	100	50
2,3,3',5,5'-PeCB	111	1000	100	50
2,3,3',5,6-PeCB	112	1000	100	50
2,3,3',5',6-PeCB	113	1000	100	50
2,3,4,4',5-PeCB[3]	114	500	50	25
2,3,4,4',6-PeCB	115	1000	100	50
2,3,4,5,6-PeCB	116	200	20	10
2,3,4',5,6-PeCB	117	200	20	10
2,3',4,4',5-PeCB[2,3]	118	500	50	25
2,3',4,4',6-PeCB	119	500	50	25
2,3',4,5,5'-PeCB	120	500	50	25
2,3',4,5,'6-PeCB	121	500	50	25
2',3,3',4,5-PeCB	122	500	50	25
2',3,4,4',5-PeCB[3]	123	500	50	25
2',3,4,5,5'-PeCB	124	1000	100	50
2',3,4,5,6'-PeCB	125	500	50	25
3,3',4,4',5-PeCB[2,3]	126	500	50	25
3,3',4,5,5'-PeCB	127	1000	100	50
Hexachlorobiphenyls				
2,2',3,3',4,4'-HxCB[2]	128	500	50	25
2,2',3,3',4,5-HxCB	129	500	50	25
2,2',3,3',4,5'-HxCB	130	500	50	25
2,2',3,3',4,6-HxCB	131	500	50	25
2,2',3,3',4,6'-HxCB	132	500	50	25
2,2',3,3',5,5'-HxCB	133	500	50	25
2,2',3,3',5,6-HxCB	134	500	50	25
2,2',3,3',5,6'-HxCB	135	500	50	25
2,2',3,3',6,6'-HxCB	136	200	20	10
2,2',3,4,4',5-HxCB	137	1000	100	50
2,2',3,4,4',5'-HxCB[2]	138	500	50	25
2,2',3,4,4',6-HxCB	139	500	50	25
2,2',3,4,4',6'-HxCB	140	500	50	25
2,2',3,4,5,5'-HxCB	141	200	20	10
2,2',3,4,5,6-HxCB	142	1000	100	50
2,2',3,4,5,6'-HxCB	143	500	50	25
2,2',3,4,5',6-HxCB	144	500	50	25

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) Con't

CB Congener[1]	IUPAC Number	Water (pg/L)	Other (ng/kg)	Extract (pg/µL)
2,2',3,4,6,6'-HxCB	145	1000	100	50
2,2',3,4',5,5'-HxCB	146	500	50	25
2,2',3,4',5,6-HxCB	147	500	50	25
2,2',3,4',5,6',-HxCB	148	1000	100	50
2,2',3,4',5',6-HxCB	149	500	50	25
2,2',3,4',6,6'-HxCB	150	1000	100	50
2,2',3,5,5',6-HxCB	151	500	50	25
2,2',3,5,6,6'-HxCB	152	1000	100	50
2,2',4,4',5,5'-HxCB[2]	153	500	50	25
2,2',4,4',5',6-HxCB	154	500	50	25
2,2',4,4',6,6'-HxCB	155	1000	100	50
2,3,3',4,4',5-HxCB[3]	156	500	50	25
2,3,3',4,4',5'-HxCB[3]	157	500	50	25
2,3,3',4,4',6-HxCB	158	200	20	10
2,3,3',4,5,5'-HxCB	159	1000	100	50
2,3,3',4,5,6-HxCB	160	500	50	25
2,3,3',4,5',6-HxCB	161	1000	100	50
2,3,3',4',5,5'-HxCB	162	1000	100	50
2,3,3',4',5,6-HxCB	163	500	50	25
2,3,3',4',5',6-HxCB	164	500	50	25
2,3,3',5,5',6-HxCB	165	1000	100	50
2,3,4,4',5,6-HxCB	166	500	50	25
2,3',4,4',5,5'-HxCB[3]	167	500	50	25
2,3',4,4',5',6-HxCB	168	500	50	25
3,3',4,4',5,5'-HxCB[2,3]	169	500	50	25
Heptachlorobiphenyls				
2,2',3,3',4,4',5-HpCB[2]	170	500	50	25
2,2'3,3',4,4',6-HpCB	171	1000	100	50
2,2',3,3',4,5,5'-HpCB	172	1000	100	50
2,2',3,3',4,5,6-HpCB	173	1000	100	50
2,2',3,3',4,5,6'-HpCB	174	500	50	25
2,2',3,3',4,5',6-HpCB	175	1000	100	50
2,2',3,3',4,6,6'-HpCB	176	1000	100	50
2,2',3,3',4',5,6-HpCB	177	500	50	25
2,2',3,3',5,5',6-HpCB	178	500	50	25
2,2',3,3',5,6,6'-HpCB	179	500	50	25
2,2',3,4,4',5,5'-HpCB[2]	180	500	50	25
2,2',3,4,4',5,6-HpCB	181	1000	100	50
2,2',3,4,4',5,6'-HpCB	182	1000	100	50
2,2',3,4,4',5',6-HpCB	183	1000	100	50
2,2',3,4,4',6,6'-HpCB	184	1000	100	50
2,2',3,4,5,5',6-HpCB	185	1000	100	50
2,2',3,4,5,6,6'-HpCB	186	1000	100	50
2,2',3,4',5,5',6-HpCB[2]	187	500	50	25
2,2',3,4',5,6,6'-HpCB	188	500	50	25
2,3,3',4,4',5,5'-HpCB[3]	189	500	50	25
2,3,3',4,4',5,6-HpCB	190	500	50	25
2,3,3',4,4',5',6-HpCB	191	1000	100	50
2,3,3',4,5,5',6-HpCB	192	1000	100	50
2,3,3',4',5,5',6-HpCB	193	500	50	25

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) Con't

CB Congener[1]	IUPAC Number	Water (pg/L)	Other (ng/kg)	Extract (pg/µL)
Octachlorobiphenyls				
2,2',3,3',4,4',5,5'-OcCB	194	500	50	25
2,2',3,3',4,4',5,6-OcCB[2]	195	1000	100	50
2,2',3,3',4,4',5,6'-OcCB	196	1000	100	50
2,2',3,3',4,4',6,6'-OcCB	197	1000	100	50
2,2',3,3',4,5,5',6-OcCB	198	500	50	25
2,2',3,3',4,5,5',6'-OcCB	199	500	50	25
2,2',3,3',4,5,6,6'-OcCB	200	1000	100	50
2,2',3,3',4,5',6,6'-OcCB	201	1000	100	50
2,2',3,3',5,5',6,6'-OcCB	202	1000	100	50
2,2',3,4,4',5,5',6-OcCB	203	1000	100	50
2,2',3,4,4',5,6,6'-OcCB	204	1000	100	50
2,3,3',4,4',5,5',6-OcCB	205	1000	100	50
Nonachlorobiphenyls				
2,2',3,3',4,4',5,5',6-NoCB[2]	206	1000	100	50
2,2',3,3',4,4',5,6,6'-NoCB	207	1000	100	50
2,2',3,3',4,5,5',6,6'-NoCB	208	1000	100	50
Decachlorobiphenyls				
DeCB[2]	209	500	50	25

[1]Abbreviations for chlorination levels:
MoCB = monochlorobiphenyl
DiCB = dichlorobiphenyl
TrCB = trichlorobiphenyl
TeCB = tetrachlorbiphenyl
PeCB = pentachlorobiphenyl
HxCB = hexachlorobiphenyl
HpCB = heptachlorobiphenyl
OcCB = octachlorobiphenyl
NoCB = nonachlorobiphenyl
DeCB = decachlorobiphenyl

[2]National Oceanic and Atmospheric Administration (NOAA) Congener of Interest.

[3]World Health Organization (WHO) Toxic Congener.

NOTE: The values in these tables are quantitation limits, not absolute detection limits. The amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to be simply detected above the background noise. The quantitation limits in these tables are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

These CRQL values are based on the analysis of samples according to the specifications given in the CB congeners SOW. Sample data are reported on a dry weight basis for all non-aqueous samples [except tissues which are reported on a wet weight basis, along with their Percent Lipid (% Lipid) content].

Table 2. Methods and Instruments

Matrix	Preparation Method	Analytical Instrument
Water	**Using Solid-Phase Extraction (SPE):** Vacuum-filtered through glass fiber on top of an SPE disk; fiber and disk extraction. **Using Separatory Funnel Extraction (SFE):** Filtrate extraction with methylene chloride using a separatory funnel. **Using Continuous Liquid-Liquid Extraction (CLLE):** Filtrate extraction with methylene chloride using a continuous liquid-liquid extractor.	HRGC/HRMS analysis
Soil/Sediment	Extraction with toluene in a Soxhlet/Dean-Stark (SDS) extractor.	HRGC/HRMS analysis
Non-Human Tissue	Extraction with methylene chloride using an SDS extractor for 18-24 hours.	HRGC/HRMS analysis

Table 3. Quality Control (QC)

QC Operation	Frequency
ANALYSES	
High Resolution Mass Spectrometry (HRMS) System Tune	Prior to the analysis of calibration standards, Initial Precision and Recovery (IPR) standards, samples, and blanks within each 12-hour period.
Window-Defining Mixture (WDM)	Following the HRMS system tune and at the beginning of each 12-hour period for each instrument used for analysis during which standards or samples are analyzed; precedes initial calibration, continuing calibration, and whenever adjustments or instrument maintenance activities are performed that may affect Retention Times (RTs).
Initial Calibration	Upon contract award, during initial setup of each instrument used prior to analysis of samples and required blanks, and each time calibration verification fails to meet the technical acceptance criteria.
Continuing Calibration Verification (CCV): Mid-Point Calibration Standard (CS3) Relative Response (RR) and Relative Response Factor (RRF)	At the beginning of each 12-hour period after the HRMS system tune during which sample data are collected and at the end of each 12-hour period.
STANDARDS	
Internal Standards	Added to all extracts prior to analysis.
Clean-Up Standard	Added to all extracts prior to cleanup.
Labeled Toxic and Level of Chlorination (LOC) Chlorinated Biphenyls (CBs)	Added to all samples including blanks, LCSs, and PE samples prior to extraction.

Table 3. Quality Control (QC) Con't

QC Operation	Frequency
BLANKS	
Performance Evaluation (PE) Samples	Prepared and analyzed (if provided) with each group of 20 field samples or less.
Laboratory Control Sample (LCS)	Prepared and analyzed with each group of 20 field samples or less of a similar matrix in an SDG.
Method Blank	Prepared and analyzed with each group of 20 field samples or less, or each time samples are extracted.
INSTRUMENT CALIBRATION	
Gel Permeation Chromatography (GPC) Calibration **NOTE:** Optional for water extracts only; should be used for soil/sediment and non-human tissue	Verified after every 20 extracts.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by ASB and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory evaluations, electronic data audits, data package audits, HRGC/HRMS tape audits, and evaluates laboratory performance through the use of blind Performance Evaluation (PE) samples.

CONTACTING EPA

For more information, or to submit suggestions to improve this analytical service, please contact:

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